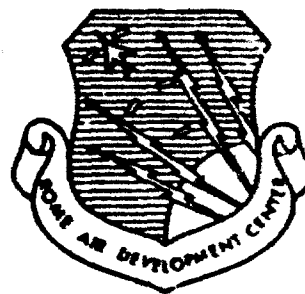


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# OPTICAL TRANSMITTER TECHNIQUES

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September 1965

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## OPTICAL TRANSMITTER TECHNIQUES


## FOREWORD


This report was prepared by Electro-Optical Systems, Inc., Pasadena, Calif., under USAF Contract No. AF 30(602)3440, (Project No. 4506, Task No. 450608). Work described in this report was accomplished by Drs. M. L. Bhaumik and L. J. Nugent of the Quantum Physics Division, and was directed by Mr. D. B. Bowen, Project Supervisor. Mr. Frank J. Rehm (EMATA) was the Rome Air Development Center project engineer.

The report describes work accomplished between 15 July and 15 October 1964.

The secondary report number assigned by Electro-Optical Systems, Inc., is 5180-Q-2.

This report has been reviewed and is approved.

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## ABSTRACT

This report describes work directed toward the design and fabrication of a gas laser oscillator and a solid-state chelate laser operating at wavelengths ranging from 5.0 to 0.3 microns. During the reporting period, the optimized material using sensitized fluorescence was tested for laser action. Under the high energy intensities of flashes, photo-reaction products are detected. This problem has been solved using new sensitizers and/or new solvents. But the quantum field is somewhat low with these sensitizers. Work is in progress to improve the quantum field. The mechanisms of the energy transfer in chelates have been further clarified and it is shown that the optical pumping scheme in the rare-earth chelate to achieve population inversion does not involve any inefficient process.

## CONTENTS

	<u>Page</u>
1. INTRODUCTION . . . . .	1
2. PROBLEM OF PHOTO-REACTION AND ITS SOLUTION . . . . .	2
3. MECHANISM OF ENERGY TRANSFER IN RARE-EARTH CHELATES . . . .	3
4. INSTRUMENTATION AND LASER TESTING . . . . .	6
REFERENCES . . . . .	8
APPENDIX I - THE ORIGIN OF THE MOLECULAR PHOSPHORESCENCE IN SOME EUROPIUM CHELATE SOLUTIONS . . . . .	9

## SECTION I

### INTRODUCTION

This is the second quarterly progress report to be submitted in accordance with the requirements of Contract No. AF 30(602)3440. The primary objectives of the work to be performed under this contract are the design and fabrication of a gas laser oscillator and a solid state chelate laser. The oscillator is to be operated at optical wavelengths (5.0 to 0.3 microns range) and is to produce an extremely stable, continuous wave output which will allow its use as the master oscillator in a master oscillator-power amplifier configuration. The solid state chelate laser must be capable of power amplification at optical wavelengths corresponding to those of the gas laser oscillator. It must also be capable of high-power pulsed operation. In both the oscillator and the amplifier, the physical phenomenon to be investigated to achieve these objectives is that which is known as "sensitized fluorescence," of which rare-earth chelates are an example.

As reported previously, the work to be performed on this program has been divided into two major subprojects: (1) The synthesis of special materials, measuring their spectrographic and pertinent physical and chemical properties, and analyzing their use as laser materials, and (2) providing the instrumentation necessary to make the required laser tests, performing these tests, and optimizing the system for high-power surveillance applications. In this report, these subprojects will be called "Chemical Physics" and "Instrumentation," and each will be reported on in a separate section of the document.

Important milestones passed and significant achievements attained during this reporting period include the following:

1. The optimized unchelated sensitized fluorescent material was tested for laser action. The quantum efficiency of the material was found to be drastically reduced by high intensity flashes.
2. The reason for the above reduction in quantum yield was traced to photo-reaction in the triplet state. An analysis of the triplet state photo-reaction reveals that this reaction can be minimized by selecting donors whose lowest triplet level is of the ( $\pi$ ,  $\pi^*$ ) rather than the ( $n$ ,  $\pi^*$ ) type.
3. Using several donors suggested by the above analysis the photo-reaction has actually been minimized, but the quantum efficiency of the new system is only about 10 percent instead of the 25 percent as in the previous material. Steps are now being taken to increase the quantum yield.
4. The mechanism of energy transfer in chelates were further classified and it is shown that the optical pumping scheme in the chelate does not involve any inefficient process.

## SECTION II

### PROBLEM OF PHOTO-REACTION AND ITS SOLUTION

An investigation of the source of photochemical changes reveals that the possible reason for the photodecomposition is the  $(n, \pi^*)$  nature of the lowest triplet state of the sensitizer. In larger molecules, like diacetylbenzene, there are triplet manifolds of  $(\pi, \pi^*)$  and  $(n, \pi^*)$  nature. Internal conversion between these levels is very fast and, therefore, the excitation energy quickly is transferred to the lowest triplet level before the transfer to the rare-earth ions.

Whether the lowest triplet level is  $(n, \pi^*)$  or  $(\pi, \pi^*)$  depends on the molecule and its surroundings. If the lowest triplet is indeed of the  $(n, \pi^*)$  variety, it can be concluded from general experience that the probability of photo-reaction involving the triplet state is much higher and therefore photo-reaction is most likely. The nature of photo-reaction is known to involve the participation of the solvent (a proton is extracted from the solvent by the excited triplet state of the molecule whereby its chemical nature and energy levels are altered). This is the reason that diacetylbenzene was more less stable in hydroxylic solvent like alcohol than in acetonitrile ( $\text{CH}_3\text{CN}$ ).

If, on the other hand, the lowest triplet level is of  $(\pi, \pi^*)$  variety, the photo-reactivity can be expected to be minimum. A simple test can be done to determine whether the triplet level of a particular molecule is of  $(n, \pi^*)$  or  $(\pi, \pi^*)$  nature. Usually, the lifetime of  $(n, \pi^*)$  triplets in EPA (ether, isopentane, ethanol 5:5:2) glass at  $77^\circ\text{K}$  is short (of the order of a millisecond). The lifetime of a triplet of the  $(\pi, \pi^*)$  variety is usually of the order of several seconds under similar circumstances.

Using the above criteria, it was found that the  $\alpha$ - and  $\beta$ -naphthaldehyde have lowest triplet levels of the  $(\pi, \pi^*)$  variety. These materials were used as sensitizers and were found to be fairly stable under high energy flashes, as expected. The quantum efficiency of  $\beta$ -naphthaldehyde and europium mixture did not go down after ten flashes of 2400 Joules at  $-50^\circ\text{C}$ .

However, the quantum efficiency of the mixtures using the above sensitizers were found to be nearly 10 percent as compared to 25 percent with diacetylbenzene. This may be the result of some impurity in the sensitizer.  $\alpha$ -naphthaldehyde is a liquid at room temperature and therefore an attempt was made to purify it by distillation. Some problems were encountered in distilling this material. Better methods for purifying this material is now under investigation. The amount of  $\beta$ -naphthaldehyde was not enough for purification. Delivery of a larger amount is expected soon. A search is also being made for other sensitizers with lowest triplet state of  $(\pi, \pi^*)$  variety.



### SECTION III

#### MECHANISM OF ENERGY TRANSFER IN RARE-EARTH CHELATES

Optical pumping in rare-earth chelate lasers involves the transfer of electronic energy from the chelate ligand to the rare-earth ion. The mechanism of this energy transfer should be understood to predict some of the laser properties. For example, if the energy is transferred only through an upper vibrational level of the triplet state, the lowest triplet level could act as a trap and, therefore, might prevent laser action. The results of some recent experiments to determine the mechanism of energy transfer are reported.

The energy level diagram of a chelate with trivalent europium is shown in Figure 1. Two mechanisms for the migration of energy from the ligand to the ion have been proposed for such a system:

- (1) Transfer of the singlet excitation energy of the ligand to its triplet level (by the intersystem crossing process) which is then intramolecularly transferred to the rare earth ion.<sup>1</sup>
- (2) The singlet excitation energy is directly transferred to the rare-earth ion.<sup>2</sup>

On a theoretical basis, one can expect that different chelates might have different mechanisms for the transfer. One can imagine a ligand and a rare-earth ion at infinite separation; the inter-action between them is zero and, thus, the transfer rate is zero. In this configuration, the lowest excited singlet lifetime is approximately  $10^{-9}$  sec and that of the triplet may be several seconds (in a rigid medium). As the distance between the rare-earth ion and the ligand decreases, the interaction between the ion and the excited singlet, and between the ion and the excited triplet of the ligand, increases. As a result, the rate of the intersystem crossing process, the radiative and radiationless processes of the  $T_1 \rightarrow S_0$  transition, increases and transfer to the rare-earth ion emission begins. This causes an additional shortening of the lifetime of the lowest triplet state and the lowest singlet state. If one neglects the transfer process for a moment, the lifetime of the lowest singlet and lowest triplet is expected to be from  $10^{-8}$  to  $10^{-10}$  sec and from  $10^{-3}$  to  $10^{-5}$  sec, respectively. Thus, on the basis of lifetime, the triplet mechanism is favored by a factor of  $10^5$  over the singlet mechanism. However, since one is dealing with consecutive processes, whether mechanism (1) or (2) is dominant should depend on whether the intersystem crossing process is faster or slower than the rate of the transfer from the singlet level.

Crosby, et al.,<sup>1</sup> suggested mechanism (1) from experimental evidence. They observed that when the ligand triplet is situated below certain rare-earth levels, no emission is observed from that level. This evidence was taken as indicative of an energy transfer via the triplet level.

Recently, Kleiner<sup>2</sup> has questioned the triplet mechanism and has pointed out that the evidence presented by Crosby, et al., in support of the triplet theory is not acceptable. He proposed that the absence of sensitized ion emission observed by Crosby, et al., might be due to quenching by the ligand triplet level rather than to the absence of the intramolecular transfer process via the ligand singlet level. Kleiner also suggested that the transfer of energy in the rare-earth chelate occurs from the ligand singlet, i.e., by mechanism (2). As yet there is no convincing proof of either mechanism.

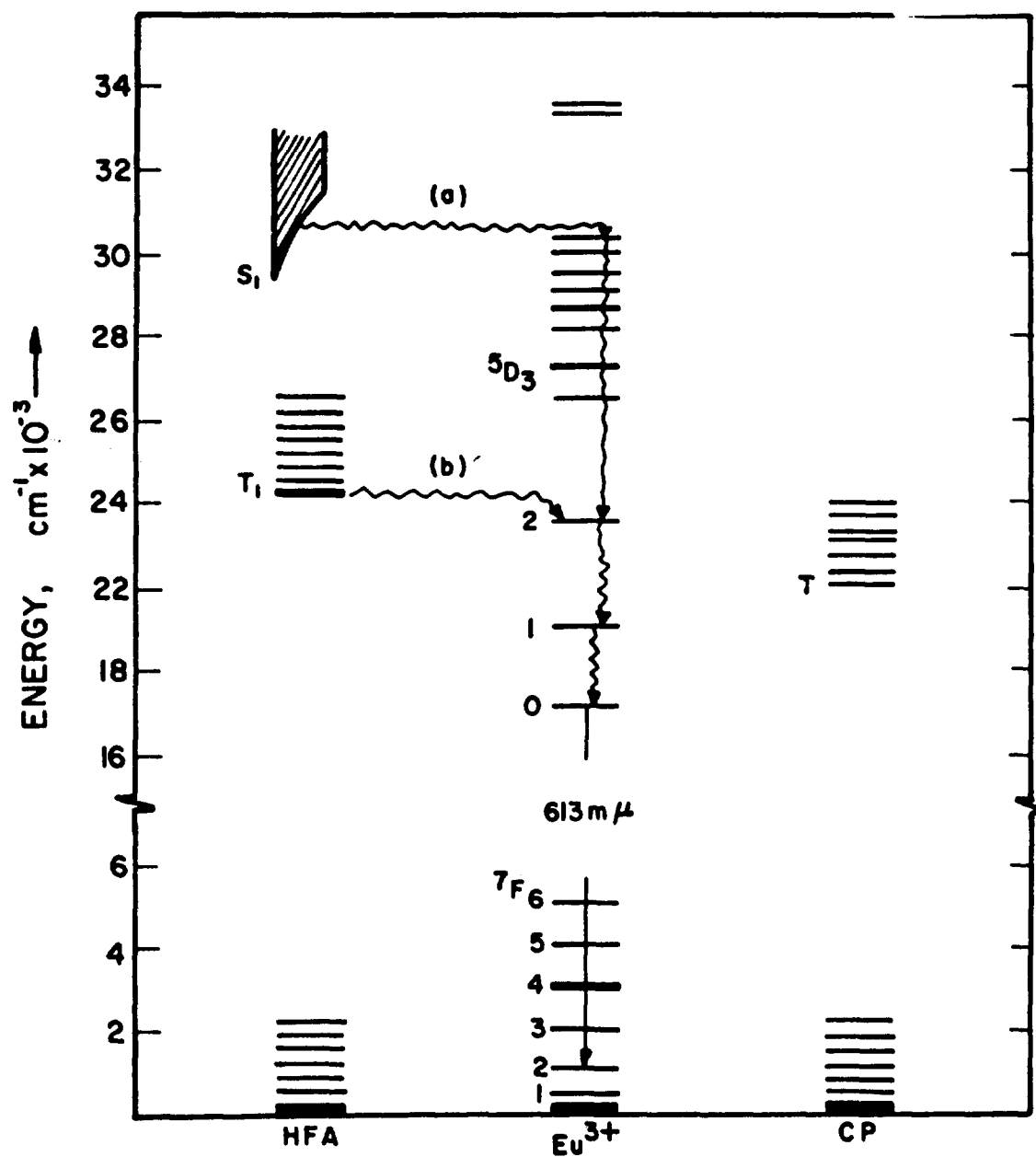


FIGURE 1. ENERGY LEVEL DIAGRAM OF HEXAFLUOROACETYLACETONATE (HFA), CIS-PIPERYLENE (CP), AND  $\text{Eu}^{3+}$

El-Sayed and Bhaumik<sup>3</sup> have shown earlier that energy can be transferred via the triplet state of a chelate. Using similar techniques of triplet-triplet transfer, it is now shown that in several Eu  $\beta$ -diketone chelates, the transfer takes place predominantly via the ligand triplet state.

Cis-piperylene has a triplet between the ligand triplet and the europium emitting level in europium hexafluoroacetylacetonate (EuHFA) while it has no appreciable absorption at the chelate excitation wavelength. When cis-piperylene is added to a solution of EuHFA in EPA (ether, isopentane, ethanol, 5:5:2), the europium emission is found to be quenched with no appreciable decrease in the fluorescence life-time. Cis-piperylene is found to quench neither the unchelated europium ion fluorescence nor the fluorescence of perylene. Therefore, the only efficient processes by which the triplet quencher can attack the chelate occur via the ligand triplet. Similar results have been obtained for europium thenoyltrifluoroacetate and europium trifluorobenzoylacetonate in EPA. This directly shows that the energy migration in these chelates takes place mostly via the chelate triplet level.

Furthermore, the rate of energy transfer can also be found to be  $10^9 - 10^{10}$  liter/mole per second from the Stern-Volmer diagram obtained in the quenching experiments. It is not impossible to imagine such a high rate of transfer via the singlet level of the chelate also. The factor that determines the route is possibly the high rate (estimated to be  $10^{11}$  per sec) of singlet-triplet crossover in these compounds. This high rate may be due to the heavy atom effect and the presence of both ( $n, \pi^*$ ) and ( $\pi, \pi^*$ ) levels between which the intersystem crossing process is known<sup>4</sup> to be very efficient.

Once it is established that the energy is transferred via the triplet level in EuHFA the next task is to solve the puzzle of the molecular chelate phosphorescence which is observed together with the ion emission. This has been taken as indicative of the fact that the transfer takes place from a higher vibrational level of the ligand triplet and that the lowest triplet level could act as a trap to prevent laser action.<sup>5</sup>

Some definitive experiments have been carried out<sup>6</sup> to show that the phosphorescence comes from a dissociation product and, therefore, is not connected with the transfer of energy in the chelate. The results of these experiments have been prepared for publication under the title "The Origin of the Molecular Phosphorescence in Some Europium Chelate Solutions." A copy is attached as Appendix i.

## SECTION IV

### INSTRUMENTATION AND LASER TESTING

The new liquid laser test system was completed and tests were initiated. The temperature control and signal detection electronics were shown to work satisfactorily. The now conventional 3:1 ethanol-methanol solution of  $\text{EuB}_4\text{HP}$  was tested for laser emission at  $-150^\circ\text{C}$ ; however, the results were incomplete. A serious problem was found with operation of the new unit at low temperatures, i.e., it is very time consuming to check the liquid for bubbles once the test unit is cooled. For bubble checking, it is presently necessary to warm to room temperature in order to prevent moisture from condensing on the cell mirrors and in the cavity. In the course of this operation a bubble which may be present at low temperature could easily disappear at higher temperatures. For this reason the new unit will be most effective at temperatures not so far removed from ambient, or for those cold solutions in which bubbles are known to be no problem upon optical pumping.

Since the new sensitized europium solution is known to be optimum near ambient temperature, it was tested in the new unit at room temperature. The system is  $\text{Eu}^{3+}$  in  $\text{CD}_3\text{OD}$  with diacetylbenzene as the sensitizer. No laser emission was observed on the first flash; however, strong photo-decomposition was observed so the solution must be replaced after each flash.

The fact that laser emission was not observed is definitely not evidence that this solution will not lase. It is important to check the Q of the cavity and mirrors to determine if the optical conditions are correct for laser emission. This will be done by observing the lasing threshold of the conventional laser solution at  $-150^\circ\text{C}$  in this cavity.

It was found last year that some of the conventional laser solutions showed laser emission while others that were supposedly identical did not. Furthermore, it was found that with the passage of time, about one week, solutions gave negative results which initially showed laser emission. Therefore, in testing the new cavity it is important to know for sure that the solution will give laser emission. The only way at present in which we can be sure of this is by showing laser emission with the old cavity. This unit has been reassembled and tests are currently underway. When a liquid known to give laser emission is obtained, it will be introduced into the new unit and tests there will continue. In the meantime, the Quantum Physics Division is making independent optical alignment checks of the near confocal optics in the new liquid laser cell.

In addition, we are proceeding to find and test new solvents such as acetonitrile in which photo-decomposition is expected to be minimized. The results of this search has been described and discussed in the section on chemical physics.

The following eight solutions are the leading candidates for liquid laser emission:

1.  $\text{EuB}_4\text{HP}$  in 3:1 Ethanol-Methanol at  $-150^\circ\text{C}$  (6130 Å)

2.  $\text{EuO}_4\text{HP}$  in 3:1 Ethanol-Methanol at  $-150^\circ\text{C}$  (6120 Å)
3.  $\text{Eu}(\text{HFA})_4\text{NH}_3$  in Acetonitrile at  $0^\circ\text{C}$  (6125 Å)
4.  $\text{Eu}(\text{TTA})_4\text{NH}_3$  in Acetonitrile at  $0^\circ\text{C}$  (6125 Å)
5.  $\text{Eu}(\text{HFA})_4\text{NH}_3$  in Perfluoroacetonitrile at  $28^\circ\text{C} - 0^\circ\text{C}$ .
6.  $\text{Eu}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$  in Perfluoroacetonitrile and in Acetonitrile, sensitized with DAB, and tested between  $-30^\circ$  and  $28^\circ\text{C}$ .
7.  $\text{Eu}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$  in  $\text{CD}_3\text{OD}$  or  $\text{D}_2\text{O}$  at  $28^\circ\text{C}$  - emission occurs at 592 and at 613 mμ.
8. Any other solutions which may be found by us or by others during this period.

These solutions will be tested during the next quarter after the optical alignment of the test cavity has been ascertained.

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## APPENDIX I

### THE ORIGIN OF THE MOLECULAR PHOSPHORESCENCE IN SOME EUROPIUM CHELATE SOLUTIONS\*

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Molecular phosphorescence emission has been observed<sup>1, 2, 3, 4</sup> together with the intramolecularly sensitized  $\text{Eu}^{3+}$  emission from rigid solutions of some europium chelates. Two mechanisms have been postulated to explain the appearance of the phosphorescence, assuming that the phosphorescence originates from the same molecular species which gives rise to the ion emission. According to the first mechanism<sup>4</sup>, the intramolecular energy transfer from the triplet level is inefficient and hence the occurrence of phosphorescence in presence of the ion emission. The same observation was interpreted by the second mechanism<sup>1, 2, 3</sup>, to imply that the intramolecular energy transfer process from the ligand to the ion proceeds from an energy level which is above the zero-point level of the lowest triplet state (where phosphorescence originates); this postulate requires that the intramolecular energy transfer process competes with either triplet-triplet internal conversion or vibrational relaxation processes, which are known to take place in  $10^{-11}$  to  $10^{-12}$  seconds.

The intramolecular energy transfer process in some chelates has recently been shown<sup>5</sup> to occur in solution\*\* from the triplet state in  $\sim 10^{-10}$  sec. These results rule out the first mechanism and make the second mechanism doubtful.

This work reports the results of an investigation on the origin of the molecular phosphorescence. In europium trifluorobenzoylacetate ( $\text{EuTBA}$ ), the phosphorescence emission is shown to have a different origin from that of the intramolecularly sensitized ion emission. Whereas the latter emission originates from the chelates, the phosphorescence seems to originate from a dissociation product of the chelate (most probably from the ligand negative ion).

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† Consultant to Electro-Optical Systems

‡ Contribution No. 1756

\*\*Since neither the intensity nor the decay time of the ion emission in the compounds under investigation change greatly when going from solution to rigid medium, the mechanism and the rate of intramolecular energy transfer in a rigid medium may be expected to be the same as those in solutions.

The EuTBA is prepared by a method reported earlier<sup>6</sup>, and the analysis shows it to be a piperidine adduct of the four ligand TBA. The melting point of this compound is 168-171°C. The absorption spectra are obtained on a Cary 14 spectrometer. The emission and activation spectra are obtained on an Aminco-Kiers spectrophosphorimeter. This apparatus is provided with a rotating drum with two openings which allows alternate excitation and viewing and thus only the phosphorescence spectrum is recorded.

The following results strongly indicate that the phosphorescence and the ion emission originate from different molecular species.

1. The activation curve of the  $\text{Eu}^{3+}$  emission of  $10^{-5}$  M rigid solution of EuTBA is different from that of the molecular phosphorescence (Fig. 1A). The former is very similar to the absorption spectrum of the chelate (at  $10^{-5}$  M) and the latter is similar to the absorption and phosphorescence activation spectra of (TBA), (using solid NaOH as the base). These results strongly suggest that the molecular species that phosphoresces must be different from that giving rise to the ion emission.
2. At higher concentration ( $10^{-2}$  M), a drastic difference between the activation curves of the phosphorescence and the ion emission is observed (Fig. 1B). In the activation curve of the ion fluorescence a new peak appears at  $366 \text{ m}\mu$ , which may be attributed to a geometrical effect.\* The activation curves, decay time and the spectrum of the phosphorescence remain unchanged except for the appearance of a dip in the activation curve at about  $366 \text{ m}\mu$ , which can be explained by the filter effect of the new strong peak of the chelate. The nature of the activation curves also indicates that the relative amount of the species showing phosphorescence is high only at low concentrations of the chelate solutions.
3. Solutions of gadolinium trifluorobenzoylacetate in EPA at  $77^\circ\text{K}$  show both strong short-lived (0.003 sec.) structured phosphorescence, as well as weak long-lived (0.2 sec.) broad phosphorescence (Fig. 1C). Both types of emissions can be resolved simply by changing the speed of rotation of the drum. The latter emission has a structure, lifetime and activation curve which are similar to those of the phosphorescence from EuTBA solution at  $77^\circ\text{K}$ . These results indicate that the relatively weak long-lived phosphorescence arises whether or not the chelate gives rise to ion emission by transfer of energy and also that the phosphorescence is due to a dissociation product which is most probably a ligand negative ion (or has emission characteristics similar to it). The relatively high amount of phosphorescence at low concentrations of the chelate is also consistent with the postulate of dissociation.

Analogous conclusions can be drawn from the studies of luminescence in europium hexafluoroacetylacetate and samarium thenoyltrifluoroacetate.

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\* Due to the front surface excitation and viewing, as in the present apparatus, a weak absorption tail can make the activation curve at higher concentrations appear to be due to a strong absorption.



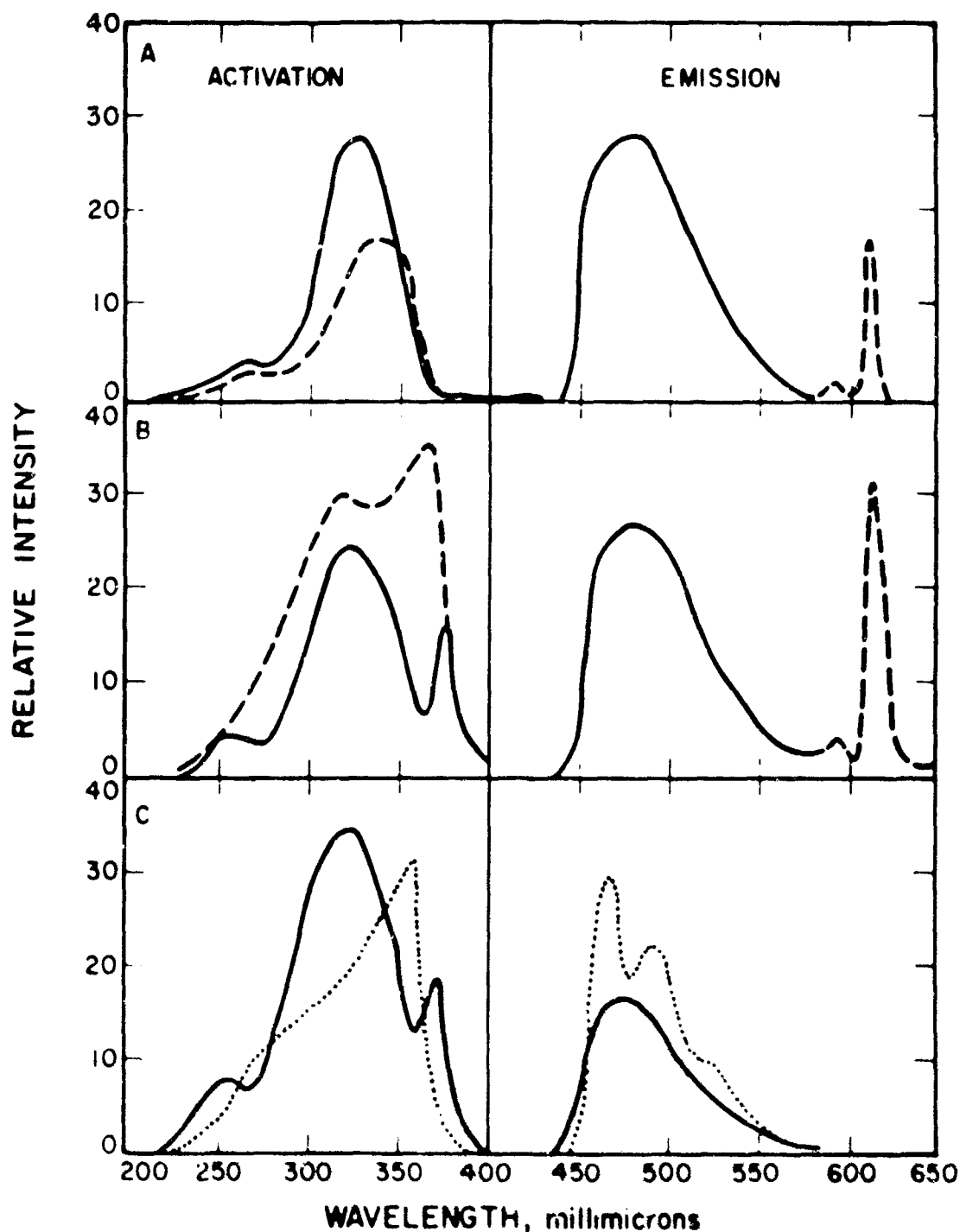


FIGURE 1 ACTIVATION AND EMISSION CURVES OF SOME TRIFLUOROBENZOYL-ACETONATE (TBA) CHELATES IN EPA AT 77°K. The chelates in A, B and C are EuTBA at  $10^{-5}$  M, EuTBA at  $10^{-2}$  and GdTBA at  $10^{-2}$  M respectively. Solid curves in A and B are due to the molecular phosphorescence and the dashed curves are for the  $\text{Eu}^{3+}$  emission. The dotted curve in C is taken with fast drum rotation in the phosphorimeter while the solid curve is recorded with a slow speed of the drum and therefore the latter represents only the long-lived phosphorescence.

We wish to acknowledge Dr. Leonard Nugent for helpful discussions.

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**13. ABSTRACT:** Enter an abstract giving a brief and factual summary of the document indicative of the report, even though it may also appear elsewhere in the body of the technical report. If additional space is required, a continuation sheet shall be attached.

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**14. KEY WORDS:** Key words are technically meaningful terms or short phrases that characterize a report and may be used as index entries for cataloging the report. Key words must be selected so that no security classification is required. Identifiers, such as equipment model designation, trade name, military project code name, geographic location, may be used as key words but will be followed by an indication of technical context. The assignment of links, rules, and weights is optional.